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IS 563 (1973): Specification for DDT, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]

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*Indian Standard*  
SPECIFICATION FOR  
DDT, TECHNICAL  
*( Second Revision )*

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*Indian Standard*  
**SPECIFICATION FOR**  
**DDT, TECHNICAL**  
*( Second Revision )*

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AMENDMENT NO. 1      DECEMBER 1980  
TO  
IS : 563-1973    SPECIFICATION FOR DDT, TECHNICAL  
( *Second Revision* )

**Alterations**

( *Page 3, clause 0.4* ) — Substitute the following for the existing clause:

‘0.4 In the preparation of this standard due consideration has been given to the provisions of the Insecticides Act, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.’

( *Page 4, Table 1* ):

- a) *Col 5, sub-heading* — Substitute ‘IS : 6940-1973\*’ for ‘IS : 6940\*’.
- b) *Note, line 3* — Substitute ‘IS : 6940-1973\*’ for ‘IS : 6940\*’.
- c) *Foot-note with ‘\*’ mark* — Delete the words ‘( Under preparation )’.

( *Page 5, clause 3.1* ) — Substitute the following for the existing clause:

‘3.1 Packing — The material shall be packed as per requirements given in IS : 8190 ( Part I )-1980\*’.

[ *Page 5, clause 3.2(g)* ] — Substitute the following for the existing matter:

‘g) The cautionary notice as worded in the Insecticides Act and Rules.’

( *Page 5, foot-note with ‘\*’ mark* ) — Substitute the following for the existing foot-note:

\*Requirements for packing of pesticides : Part I Solid pesticides (*first revision*).’

( *Page 6, clause 4.1* ) — Substitute the following for the existing clause:

‘4.1 Representative samples of the material shall be drawn as prescribed in Indian Standard Methods for sampling of pesticides and their formulations’ (*under preparation* ).

**Note** — Till such time the standard under preparation is published, samples shall be drawn as agreed to between the parties concerned.’

( *Page 6, clause 4.1.1* ) — Delete.

( *Page 6, foot-note with '\*' mark* ) — Delete the words '( Under preparation )'.

( *Page 10, clause B-3.1* ) — Delete 'f' wherever it occurs in the clause.

( *Page 10, clause B-3.1.1* ) — Delete.

( *Pages 11 and 12, clause C-3.1* ) — Delete 'f' wherever it occurs in the clause.

( *Page 12, clause C-3.1.1* ) — Delete.

( AFCDC 6 )

*Indian Standard*  
**SPECIFICATION FOR**  
**DDT, TECHNICAL**  
*( Second Revision )*

**0. F O R E W O R D**

**0.1** This Indian Standard ( Second Revision ) was adopted by the Indian Standards Institution on 18 January 1973, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

**0.2** This standard was first published in 1955 and subsequently amended to modify the method of test for moisture determination. The first revision of this standard was issued in 1961 in order to incorporate metric values in place of the FPS values and new sampling scheme in place of the existing one. Since the publication of the first revision of the standard, some observations had been made regarding the amendment of the requirements for hydrolysable chlorine content and chloral hydrate content in order to align them with the latest WHO specification for this product. Further, an amendment which was issued to the revised standard in 1963 regarding the packing requirement had also to be incorporated in the standard with certain modifications. This second revision, therefore, is aligned with the WHO specification, as far as possible, and incorporates revised packing requirements.

**0.3** DDT ( dichloro diphenyl trichloroethane ), technical, is employed in large quantities in the preparation of a number of insecticidal formulations used in the control of pests of agricultural and of public health importance. DDT, technical, has a variable composition and contains several chemical components of which the *pp'*-isomer is the active principle.

**0.3.1** DDT, the chemical name of which is 1, 1, 1-trichloro-2, 2-di (*p*-chlorophenyl)-ethane has the structural and empirical formulae, and molecular weight as given below:

<i>Empirical Formula</i>	<i>Structural Formula</i>	<i>Molecular Weight</i>
$C_{14}H_9Cl_5$		354.5

**0.4** This standard is one of a series of Indian Standards on pesticides and their formulations.

**0.5** For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960\*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

## 1. SCOPE

**1.1** This standard prescribes the requirements and the methods of test for DDT, technical.

## 2. REQUIREMENTS

**2.1** The material shall be in the form of granules, flakes or powder, free from extraneous impurities or added modifying agents and shall be white to creamy in colour.

**2.2** The material shall also comply with the requirements given in Table 1.

**TABLE 1 REQUIREMENTS FOR DDT, TECHNICAL**

SL. No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO	
			Appendix	Cl No. in IS : 6940*
(1)	(2)	(3)	(4)	(5)
i)	<i>pp'</i> -isomer content, percent by mass, <i>Min</i>	70.0	A	—
ii)	Setting point, °C, <i>Min</i>	89	—	8
iii)	Melting point of separated <i>pp'</i> -isomer, °C, <i>Min</i>	104	—	6
iv)	Total organic chlorine content, percent by mass	49.0 to 51.0	B	—
v)	Hydrolysable chlorine content, percent by mass	9.5 to 11.0	C	—
vi)	Matter insoluble in acetone, percent by mass, <i>Max</i>	1.0	—	9
vii)	Acidity (as $H_2SO_4$ ), percent by mass, <i>Max</i>	0.9	—	11.3
viii)	Moisture, percent by mass, <i>Max</i> ( <i>see Note</i> )	1.0	—	4.1 or 4.2

**NOTE** — The moisture content shall be determined by the Karl Fischer method, or where practicable by the Dean and Stark method as per clauses 4.1 and 4.2 respectively of IS : 6940\*.

\*Methods of tests for pesticides and their formulations. (Under preparation).

\*Rules for rounding off numerical values (revised).

### 3. PACKING AND MARKING

**3.1 Packing** — The material shall be packed in one of the following ways:

- Double hessian bags with a polyethylene liner of minimum 0.037 mm (150 gauge) thickness;
- Double hessian bags 310 g on both sides interlined with 60 g kraft paper bounded with 90 g/m<sup>2</sup> bitumen on each run;
- Containers made of wood with polyethylene liner;
- Fibreboard;
- Tinplate;
- Mild steel; and
- High density polyethylene (HDP) woven sacks (see IS : 6340-1971\*) with integrated lining of low density polyethylene of 0.037 mm (150 gauge) thickness.

**3.2 Marking** — The containers shall be securely closed and shall bear legibly and indelibly the following information in addition to the provisions as required under the Insecticides Act and Rules:

- Name of the material;
- Name of the manufacturer;
- Date of manufacture;
- Batch number;
- Net mass of contents;
- pp'*-isomer content, percent by mass; and
- The minimum cautionary notice worded as under :  
'KEEP WELL AWAY FROM FOODSTUFFS, EMPTY FOODSTUFF CONTAINERS AND ANIMAL FEED.'

**3.2.1** When hessian bags are used for packing this material, the pictorial marking for 'USE NO HOOKS. DO NOT PUNCTURE' as specified in IS : 1286 - 1967† shall be stencilled.

**3.2.2** The containers may also be marked with the ISI Certification Mark.

**Note** — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

\*Specification for high density polyethylene woven sacks.

†Pictorial markings for handling of goods in general (first revision).

#### 4. SAMPLING

**4.1** Representative samples of the material shall be drawn as prescribed in IS : 6940\*.

**4.1.1** Following a given order for the production of the material, the purchaser, if he so desires, shall have the right to draw samples of the material during manufacture of each batch at the time of packing the material in the containers before sealing them. Such samples too shall be drawn as prescribed in IS : 6940\*.

#### 5. TESTS

**5.1** Tests shall be carried out by the appropriate methods referred to in col 5 of Table 1.

**5.2 Quality of Reagents** — Unless specified otherwise, pure chemicals and distilled water (*see IS : 1070 - 1960†*) shall be employed in tests.

**NOTE** — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

### A P P E N D I X A

[ *Table 1, Item (i)* ]

#### DETERMINATION OF *pp'*-ISOMER CONTENT

##### A-1. REAGENTS

**A-1.1 Ethyl Alcohol** — Aqueous, 75 percent by volume.

**A-1.2 Saturated Solution of Pure *pp'*-isomer of DDT** — Make a paste of 100 g of the material with 50 ml of rectified spirit (*see IS : 323-1959‡*), 95 percent by volume. Add to this paste 300 ml of water and mix. Filter the mixture through a Buchner funnel. Transfer the residue to a 600-ml beaker, add 250 ml of rectified spirit, 95 percent by volume, and mix the contents. Chill the contents of the beaker in a mixture of salt and ice, and filter. Wash the residue with 100 ml of cold rectified spirit, 95 percent by volume. Repeat the procedure with the residue using petroleum ether (B.P. 40 to 60°C) instead of rectified spirit. Dry the residue obtained from petroleum ether extraction (expected yield 65 g). Recrystallize the dry residue from 800 ml of hot rectified spirit, 95 percent by volume (expected

\*Methods of tests for pesticides and their formulations. (Under preparation)

†Specification for water, distilled quality (*revised*).

‡Specification for rectified spirit (*revised*).

yield 60 g.). Repeat the extraction with petroleum ether and recrystallization, from hot rectified spirit until the final product on drying gives a melting point between 110°C and 110.5°C. (Usually two more extractions will be necessary and the final yield will be about 46 g.)

Prepare a saturated solution of the pure *pp'*-isomer of DDT in ethyl alcohol and store this solution in a thermostatically controlled bath at 25.0  $\pm$  0.5°C.

## A-2. PROCEDURE

**A-2.1** Weigh accurately about 2 g of the material into a 250- to 300-ml Erlenmeyer flask, add to it 150 ml of saturated solution of pure *pp'*-isomer of DDT at 25.0  $\pm$  0.5°C and mix. Fit the flask with a reflux condenser and reflux the contents of the flask until the whole of the material is completely dissolved. Remove the reflux condenser, stopper the flask and allow it to cool slowly in the air to about 26 to 30°C, when crystals of *pp'*-isomer of DDT separate out. If separation of oil occurs at this stage, redissolve the oil by refluxing the contents of the flask again and, if necessary, add a seeding crystal of pure *pp'*-isomer of DDT during cooling.

**A-2.2** Place the flask and contents in the thermostatically controlled bath 25.0  $\pm$  0.5°C for four hours shaking the contents of the flask intermittently. Filter the crystals with suction through a tared Gooch crucible containing a disc of filter paper, taking care that as little air as possible is sucked through the wet crystals during filtration. Dry the crucible and its contents to constant weight at 78 to 80°C, cool in a desiccator, weigh and find out the mass of the dry crystals.

**A-2.3** Reserve a portion of the dry crystals of *pp'*-isomer of DDT (see A-2.2) for the determination of its melting point.

## A-3. CALCULATION

**A-3.1** Calculate the *pp'*-isomer content in the material by the following formula adding empirical correction of 1.4 percent to give results in agreement with known mixtures:

$$\text{pp'-isomer content of the material, percent by weight} = 1.4 + \frac{100 m}{M}$$

where

$m$  = mass in g of the dry crystals of *pp'*-isomer (see A-2.2), and

$M$  = mass in g of the material taken for the test (see A-2.1).

## APPENDIX B

### [ *Table 1, Item (iv)* ]

## DETERMINATION OF TOTAL ORGANIC CHLORINE CONTENT

### B-1. REAGENTS

**B-1.1 Benzene** — free from thiophene and chlorine.

**B-1.2 Isopropyl Alcohol** — of 99 and 50 percent concentration by volume.

**B-1.3 Metallic Sodium** — pure, in the form of ribbon or cut in small pieces.

**B-1.4 Phenolphthalein Indicator Solution** — one percent (*m/v*) in rectified spirit (*see IS : 323-1959\**).

**B-1.5 Dilute Nitric Acid** — 50 percent by volume.

**B-1.6 Standard Silver Nitrate Solution** — 0.1 N.

**B-1.7 Ferric Ammonium Sulphate Solution** — saturated, aqueous, freshly prepared.

**B-1.8 Standard Potassium Thiocyanate Solution** — 0.1 N.

### B-2. PROCEDURE

#### B-2.1 Determination of Total Chlorine Content

**B-2.1.1** Weigh accurately about 1 g of the material, transfer it to a 250-ml graduated flask, add 10 ml of benzene to dissolve the material and then make up the volume with 99 percent isopropyl alcohol. Transfer a 25-ml aliquot to a 250-ml Erlenmeyer flask.

**B-2.1.2** Introduce approximately 2.5 g of metallic sodium into the Erlenmeyer flask containing the aliquot. Connect the flask to a reflux condenser and boil gently for at least half an hour, shaking the flask occasionally. Dissolve the excess metallic sodium by cautiously adding 10 ml of 50 percent isopropyl alcohol through the condenser at the rate of one to two drops per second. Boil for an additional 10 minutes and then add 60 ml of distilled water.

**B-2.1.3** Cool, then add 2 to 3 drops of phenolphthalein indicator solution. Neutralize by adding dilute nitric acid dropwise and add 10 ml in excess. If necessary, cool the flask to room temperature, and add a known volume of the standard silver nitrate solution in slight excess and coagulate the

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\*Specification for rectified spirit (*revised*).

precipitated silver chloride by digesting on a steam-bath for half hour, with frequent stirring. Cool the flask and, if necessary, filter the contents of the flask through a fast qualitative filter paper, wash thoroughly with distilled water, collecting the filtrate quantitatively in a conical flask. Add 5 ml of ferric ammonium sulphate solution either to the cooled unfiltered mixture or to the filtrate, as the case may be, and titrate the excess of the silver nitrate with the standard potassium thiocyanate solution. (The end point is the appearance of red ferric thiocyanate colour.)

**B-2.1.4** Carry out a blank determination on the reagents, using the method given under **B-2.1.1** to **B-2.1.3**.

### **B-2.2 Determination of Inorganic Chlorine Content**

**B-2.2.1** Test the material for the presence of chloride ions by the method given under **B-2.2.2**. In case free chloride ions are detected, quantitatively determine the percentage by mass of inorganic chlorine in the material by the method given under **B-2.2.3**.

**B-2.2.2 Qualitative Test for Chlorides** — Shake about 1 g of the material in the form of a fine powder with 5 ml of water and filter. To the clean filtrate, add a few drops of concentrated nitric acid (see IS : 264-1968\*) and 1 ml of silver nitrate solution (approximately two percent *m/v*). If a white turbidity or opalescence appears, quantitatively determine the inorganic chlorine in the material as given in **B-2.2.3**.

**B-2.2.3** Weigh accurately about 10 g of the material in the form of a fine powder into a 250-ml beaker and stir with a small quantity of water. Add more water, allow to stand and filter by decantation through a quantitative filter paper, collecting the filtrate in another beaker. Repeat extraction with water and filtering until a few drops from the tail of the funnel do not give a test for chlorides. Add 5 ml of concentrated nitric acid (see IS : 264-1968\*) to the combined filtrate contained in the beaker and heat it to about 50°C. Add to the hot filtrate sufficient volume of silver nitrate solution (5 percent *m/v*). Boil to coagulate the precipitated silver chloride. Protect the precipitated silver chloride, by wrapping black paper around the container. Cool the contents of the beaker and filter through a tared Gooch crucible or sintered glass crucible (G No. 4). Wash the precipitate first with dilute nitric acid (approximately 4 N) and then with cold water. Dry the crucible and its contents to constant weight at 130 ± 2°C and find the mass of the silver chloride. Determine the percentage by mass of inorganic chlorine in the material from the formula:

$$c = \frac{24.74 m}{M}$$

\*Specification for nitric acid (first revision).

where

$c$  = inorganic chlorine content of the material, percent by mass;

$m$  = mass in g of the silver chloride; and

$M$  = mass in g of the material taken for the test.

(Alternatively, the inorganic chlorine content of the material may be determined by a volumetric method.)

### B-3. CALCULATION

**B-3.1** Total organic chlorine content of the material,  $= \frac{35.46 (V-v)Nf}{M} - c$ , percent by mass

where

$V$  = volume in ml of the standard potassium thiocyanate solution required for the blank determination (see B-2.1.4);

$v$  = volume in ml of the standard potassium thiocyanate solution required for the test with material (see B-2.1.3);

$N$  = normality of the standard potassium thiocyanate solution;

$f$  = correction factor (see B-3.1.1);

$M$  = mass in g of the material taken for the test (see B-2.1.1); and

$c$  = inorganic chlorine content of the material, percent by mass (see B-2.2).

**B-3.1.1 Correction Factor** — Determine the value of the correction factor  $f$  from the formula:

$$f = \frac{T}{A}$$

where

$T$  = percent by mass of total chlorine in the DDT, calculated theoretically, which is equal to 50.01; and

$A$  = actual value, percent by mass of total chlorine in pure, recrystallized DDT, determined by the method prescribed in B-2.1.

**Note** — The correction factor  $f$  includes the error arising from the impurities of the reagents, the method itself and the handling of the method in a given laboratory. Its value shall be within the range of 0.98 to 1.02.

## APPENDIX C

[ *Table 1, Item (v)* ]

## DETERMINATION OF HYDROLYSABLE CHLORINE CONTENT

## C-1. REAGENTS

## C-1.1 Acetone

C-1.2 Alcoholic Potassium Hydroxide Solution — 1 N.

C-1.3 Dilute Nitric Acid — approximately 2 N.

C-1.4 Standard Silver Nitrate Solution — 0.1 N.

C-1.5 Ferric Ammonium Sulphate Solution — saturated, aqueous, freshly prepared

C-1.6 Standard Potassium Thiocyanate Solution — 0.1 N.

## C-2. PROCEDURE

C-2.1 Weigh accurately about 0.5 g of the material into a 250-ml Erlenmeyer flask. Add to it 50 ml of acetone and 20 ml of alcoholic potassium hydroxide solution. Keep it at 20 to 25°C for 15 minutes and then add 50 ml of water. Add to the contents of the flask 20 ml of dilute nitric acid and exactly 25 ml of the standard silver nitrate solution. Coagulate the precipitate of silver chloride by digesting on a steam-bath for half an hour, with frequent stirring. Cool the flask and, if necessary, filter the contents of the flask through a fast qualitative filter paper collecting the filtrate quantitatively in a conical flask. Add 5 ml of ferric ammonium sulphate solution either to the cooled unfiltered mixture or to the filtrate, as the case may be, and titrate the excess of the silver nitrate with the standard potassium thiocyanate solution. (The end point is the appearance of red ferric thiocyanate colour.)

C-2.2 Carry out a blank determination on the reagents using the method given under C-2.1.

## C-3. CALCULATION

C-3.1 Hydrolysable chlorine content of the material,  $\frac{3.546 (V-v) Nf}{M} - c$   
percent by weight

where

$V$  = volume in ml of the standard potassium thiocyanate solution required for the blank determination ( *see C-2.2* );

$v$  = volume in ml of the standard potassium thiocyanate solution required for the test with the material (see C-2.1);  
 $N$  = normality of the standard potassium thiocyanate solution;  
 $f$  = correction factor (see C-3.1.1);  
 $M$  = mass in g of the material taken for the test (see C-2.1);  
and  
 $c$  = inorganic chlorine content of the material, percent by mass as determined under C-2.2.

**C-3.1.1 Correction Factor** — Determine the value of the correction factor  $f$  from the formula:

$$f = \frac{T}{A}$$

where

$T$  = percent by mass of hydrolysable chlorine in pure DDT, calculated theoretically, which is equal to 10.00; and  
 $A$  = actual value, percent by mass, of hydrolysable chlorine in pure, recrystallized DDT, determined by the method prescribed in C-2.1.

**NOTE** — The correction factor includes the error arising from the impurities of the reagents, the method itself and the handling of the method in a given laboratory. Its value shall be within the range of 0.98 to 1.02.

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